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Sincerely,

## **FINAL REPORT**

### **Electro-Optics Based on Novel Materials Modifications**

**ARO Contract Number DAAG55-98-C-0045**

**Period covered: 1 July 1998 through 31 Dec 2000**

**Performed at: Charles Evans and Associates**

**Principal Investigators: Drs. C.A. Evans, Jr. and R.G. Wilson**

### **Introduction**

This report describes work done and results obtained from 1 July 1998 through 31 Dec 2000. The report is divided into sections by technical area.

Aims or goals of this work included growth, doping, and characterization of new materials of interest to ARO for electro-optics and electronics applications within the DoD, often involving support for university research through collaborative programs initiated by Dr. John Zavada. A concomitant goal is the publication of papers that describe the results of this work and these collaborations.

The principal investigators for this program were Dr. C.A. Evans, Jr. of Charles Evans and Associates, and Dr. R.G. Wilson, Consultant.

### **Summary of work carried out, and results**

#### **III-Nitrides**

Research related to the growth, characterization, and fabrication of devices in III-nitrides, including GaN, AlN, GaAlN, InGaN, and InAlN, are intended to be a significant effort on this contract. III-nitrides are currently of great interest for both optical and electronic devices and applications, including blue, green, and violet LEDs and high power/high temperature electronics (microelectronics, especially microwave). Our contributions in this field include ion implantation for doping and co-doping (Mg, Ca, P, C, Si, Er, Pr) and for

impurity characterization using SIMS (H, C, O, Si); SIMS measurements of both intentional and unintentional impurities -- both concentration and depth profiles.

Photoluminescence measurements are another important area of work in these nitrides. Band edge measurements are being made at Kansas State Univ. (Prof. X. Jiang) and longer wavelength measurements are being made at Hampton University (Prof. U. Hoemmerich). Related papers are listed in the last section on Publications. Figure 4 shows a representative PL measurement of band edge luminescence (Kansas State Univ.) and Fig. 5 shows a representative PL measurement of 1.54 $\mu$ m stimulated radiation (Hampton Univ.).

### **GaN and rare earth implantation**

III-nitrides, and GaN in particular, have become important for high temperature and high voltage devices and for optical devices, including all color LEDs and lasers. AlN and AlGaN are used in these devices. Many laboratories are working on the growth and doping of device structures using III-nitrides. Because of this and our involvement in this work, we continued to improve our SIMS analysis capability for III-nitrides. We continued to prepare ion implanted standards for SIMS analyses and to improve SIMS analysis techniques for III-nitrides using these standards. Because GaN is often grown on sapphire substrates (when not on SiC), which are good insulators, the best SIMS technology was often in the past to use a quadrupole instrument. We improve the technology for using CAMECA sector magnet SIMS equipment, which will improve quantification, consistency, increased sputtering rate (an advantage for deep analyses), and allow the use of high mass resolution. We spent considerable time implanting SIMS standards and studying SIMS analysis approaches in this work. An important result is that this work has provided a good SIMS analysis capability for III-nitrides at Charles Evans and Associates. Government programs such as this ARO contract benefit from this SIMS analysis capability.

We implanted various GaN materials with Er and Pr, and also with oxygen to act as a co-activator, although we stopped this latter process during the year (because we determined that sufficient oxygen exists in these materials as-grown). We annealed some of these samples, and sent all of them on to Kansas State and Hampton Univ. for band edge luminescence and for stimulated PL emission measurements at 1.5 or 1.3  $\mu$ m, for Er and Pr, respectively. The results of these measurements were sent to John Zavada, who keeps the various involved people informed.

We prepared Si 'witness' samples of our implants of Er and Pr for SIMS measurements of the depth profiles of Er or Pr, and for RBS measurements of the implanted Er or Pr as checks on the implantation process. This is critical because implants of Er and especially Pr are very difficult and often contain impurities in the ion beam at masses close to those of Er and Pr that are the result of the high required temperature that causes Fe

chlorides from the ion source, and from prior implants of elements that also combine with Cl in the ion source, or have interfering molecular ions in the mass range from 135 to 175. We spend a significant effort performing SIMS and RBS measurements on these samples, and re-implanting samples when these measurements show that the implants are not what was intended.

We mounted pieces of GaN from UTA, KSU, and UFL, plus witness Si for Pr implantation, and attempted to implant Pr - unsuccessfully once; with what turned out to be a low fluence of Pr on the second attempt; and with what turned out to be the desired fluence ( $5.7 \times 10^{13} \text{ cm}^{-2}$ ) on the third attempt. These Pr-implanted samples of GaN were studied at Kansas State and Hampton Universities for band edge and infrared PL in conjunction with fairly high annealing temperatures. Now that the optical testing of these samples is completed, we will measure (early 2001), using SIMS, the depth profiles of Pr to determine whether any redistribution of Pr has occurred during the associated annealing. The quality of these implants was determined using both SIMS and RBS measurements, made in our laboratory.

Certain rare earth elements emit light of specific wavelengths under electrical or optical stimulation from essentially any matrix into which they are introduced. The presence of oxygen in some host materials is known to enhance the intensity of this emission. Important examples of these elements are Er and Pr because of the application to optical communication systems, namely  $1.5 \mu\text{m}$  for Er and  $1.3 \mu\text{m}$  for Pr. We pursued the development of an implantation capability for Er and Pr and implanted these elements in a variety of materials. This technology for Er and Pr is now well established, but implanting Pr proved to be more difficult. The problems were solved by using a separate ion source dedicated to rare earth elements.

In collaboration with Professor Steve Bishop of University Illinois, we studied Mg- and Er-doped GaN for Mg, Er, and other elements using SIMS and calibrated using implanted standards in the same material. We measured the density of Mg and of Er in several samples on GaN. We implanted Mg and Er into samples of material that University of Illinois had been studying for photoluminescence (PL) emission. We determined the doping level of Mg in Er-implanted Mg-doped GaN. We implanted Er in some GaN. We also measured the amount of C and O in these samples.

A project was initiated to study PL emission from samples of GaN doped with two rare earth elements or one rare earth element and another element. We implanted Er plus Yb, Er plus Cr, and Yb plus Cr, and Yb and Cr only. These samples were submitted for PL measurements. Some of these samples were annealed at Kansas State University at temperatures of 900, 1000, and 1100°C. We measured the depth distributions of the implanted species using SIMS.

## GaN SIMS analysis

In support of the improved SIMS analysis capability described in the preceeding section, some work with implanted SIMS standards in GaN are described here. We have implanted various elements into samples of GaN and performed various SIMS analyses using those standards. Included are:  $^1\text{H}$  and  $^2\text{H}$ ,  $^{12}\text{C}$ ,  $^{16}\text{O}$  and  $^{18}\text{O}$ ,  $^{24}\text{Mg}$ ,  $^{28}\text{Si}$  and  $^{30}\text{Si}$ ,  $^{32}\text{S}$  and  $^{34}\text{S}$ ,  $^{54}\text{Fe}$  and  $^{56}\text{Fe}$ ,  $^{58}\text{Ni}$ ,  $^{80}\text{Se}$ ,  $^{128}\text{Se}$ , and  $^{130}\text{Se}$ . All of these elements produce depth profiles, but some are better than others. Sometimes densities of impurities in nitride materials cause high background levels. This emphasizes the need to use the rarer isotopes, which are included in the above listing. We have studied and compared the relative value of different SIMS analysis approaches, namely oxygen bombardment and positive secondary ions, cesium bombardment and positive secondary ions using Cs molecules (e.g.  $\text{MgCs}^+$  and  $\text{ZnCs}^+$ ), and cesium bombardment and negative secondary ions using atomic ions and molecular ions (e.g.  $\text{SiN}^-$ ).

## Implanted dopants in GaN

We implanted Be, C, Mg, S, Se, Te into GaN (to go with Si already done), annealed samples at temperatures up to  $900^\circ\text{C}$  (CE&A/RGW) and at 1000, 1100, 1200, 1300, and  $1400^\circ\text{C}$  (Univ. Florida), then measured the resulting depth distributions using SIMS. Results: Be, C, Mg, S, Se, and Te do not redistribute for temperatures up to  $1450^\circ\text{C}$  in GaN ( $1200^\circ\text{C}$  for Be). This result is in contrast to all other materials studied so far, and demonstrates the great thermal stability of GaN and dopants in GaN. This is a significant result that has been demonstrated under this program. Several publications have put these results into the 1998/99 literature; See the list of Publications at the end of this report. This result is also in some disagreement with previous data measured and reported under this program, namely: redistribution of Be and S were observed. The explanation is probably that the quality of the early material was poor. Much improvement has been made in the last two years in the quality of GaN. Probably defects or columnar structure with associated grain boundaries existed in previous material. The material used in this 1998 set of measurements was all the same and all from EMCORE. In particular, the new S data are very nice, and the new Be data show only a slight decrease in intensity for the highest temperature, but no significant redistribution. Thus, only H shows redistribution for temperature greater than  $1000^\circ\text{C}$ , and then only really in p-type material, very little in n-type.



## **Study of behavior of hydrogen in n- and p-type GaN**

We collaborated in a study of the behavior of H in n- and p-type GaN with Univ. Florida (UFL) (Profs. S. Pearton, C. Abernathy, and F. Ren). We performed anneals at temperatures up to 900°C in a furnace. Then UFL did anneals at temperatures of 1000, 1100, and 1200°C in a special flash lamp annealer. We subsequently measured the depth distributions of H using SIMS. Results: The two sets of anneals match up to some extent, but not exactly. UFL's anneals were 10 s RTP; ours were 10-min furnace anneals -- something might be different about the two processes. Another difference to note is that there is a substrate interface shallower for the p-type than for the n-type, an interface that could be the source of defects and "gettering" of the H. The first profiles showed a pile-up of H at that interface for 800 and 900°C. The higher temp RTP anneals show no H at all. The data for the n-type material are consistent - and better. The profiles for 1000, 1100, & 1200°C are similar and fit with the lower temperatures but there is less change than might have been expected.

## **Er implanted materials**

Following is a summary list of the materials that were implanted with Er (and some with Pr) during the three years of this ARO program: diamond, Si, SiC, SiGe, Ge, AlN, GaN, GaP, GaAs, AlGaAs, InP, InAs, InSb, SiO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub>, LiNbO<sub>3</sub>, ZnSe, Al, Ni, and Au. Annealing studies were carried out on some of these materials, with SIMS measurements made to determine thermal stability and redistribution.

## **Hydrogen in Other Materials**

Studies of the properties and effects of H in III-nitride materials and other optoelectronic materials was one thrust of our research, especially in collaboration with the group at University of Florida. A summary of that work is given below.

Residual hydrogen in MOVCD-grown device structures such as heterojunction bipolar transistors, thyristors, and p-i-n diodes intended for high power, high temperature applications, that originates from the growth precursors decorates Mg-doped layers and AlGaIn/GaN interfaces. There is a significant difference in the diffusion characteristics and thermal stability of implanted H between n- and p-type GaN, caused by the stronger affinity of H to pair with acceptor dopants and possibly to the difference in H<sub>2</sub> formation probability.

Atomic H permeates GaN during various device processing steps. This indiffusion is enhanced by the presence of high defect densities in heteroepitaxial materials. SIMS

profiling after processing using deuterated chemicals (to enhance the SIMS detection sensitivity) show that H can diffuse into GaN at temperatures as low as 80°C. The primary effect of this H is passivation (electrical de-activation) of Mg acceptors in p-GaN via formation of neutral Mg-H complexes, which can be dissociated through either minority carrier (electron) injection or simple annealing. We have evidence that all acceptors in GaN, viz. C, Mg, Ca, Zn, and Cd, form complexes with H.

## **GaN device structures for LEDs and other optical devices**

We collaborated with Univ. California at Santa Barbara (UCSB) (Profs. J. Speck and S. denBaars) for characterization and rare-earth doping of GaN (and other III-nitride materials). SIMS results of GaN layers and structures received from UCSB (grad. student Monica Hansen)

We measured depth distributions of H, C, O, and Si, using SIMS, in a sample of template GaN for the subsequent growth of laser diode structures for Er pumping (implanted and or grown-in). Results: This material is very clean, for GaN. The measured densities of H, C, O, and Si were essentially the background densities or the SIMS measurement ( $10^{17}$  for C, and about  $10^{18}$  for H, O, and Si). However, the sample is not Si-doped, as was intended, unless the intended doping was less than  $5 \times 10^{17} \text{ cm}^{-2}$ .

An MBE-grown layer of Si-doped GaN grown on MOCVD GaN was studied for comparison of H, C, and O impurity concentrations - between MBE and MOCV. We carried out the same measurements for an MBE-grown Si-doped layer of GaN on an MOCVD-grown GaN layer. Results: This sample had a 60-nm surface layer doped with Si at  $1 \times 10^{20} \text{ cm}^{-2}$ . H, C, and O were all modest impurities (mid  $10^{19} \text{ cm}^{-2}$ ) in both the MBE and MOCVD layers, compared with the template material GaI.

We analyzed the LED structure for both the elements, H, C, O, and Si, and also for composition of structure. In general, the structure agreed with the intended one, but with the following differences: There was no significant Si doping of the deep GaN, AlGa<sub>x</sub>N<sub>1-x</sub> (thick), or InGa<sub>x</sub>N<sub>1-x</sub> layers, unless that doping was intended to be less than  $5 \times 10^{17}$  or  $1 \times 10^{18} \text{ cm}^{-2}$ . The In content appeared to be low; it was about 1 % of the Ga. A strong C impurity (mid to high  $10^{19} \text{ cm}^{-2}$ ) tracks the Al content.

Er was successfully implanted into pieces of the above samples along with other materials. The Er implants were verified using SIMS measurements made on a witness piece of Si implanted beside these samples, and the fluence was verified to be approximately  $5 \times 10^{13} \text{ cm}^{-2}$ . These samples were then delivered to UCSB for subsequent device layer growth and characterization.



### **3C SiC**

We carried out research on 3C SiC with both Professors A. Steckl of Univ. of Cincinnati, and Prof. James Kolodzey of Univ. of Delaware. We received a sample of 3C SiC (a film grown on a Si wafer) from post doc Cyril Guedj at Univ. Delaware - for implantation of high dose Ge alone and also plus H. These implants were performed, and the samples were returned to UDE. Some interesting results are being obtained.

### **ZnSe, ZnTe, and CdTe**

II-VI materials can have large or small bandgaps. They are of interest for a variety of applications, one of which is detectors, especially for long wavelengths. We carried out a modest investigation of some undoped and doped II-VI materials by performing materials analyses using SIMS. We determined the annealing behavior of H in ZnSe by implanting H, annealing, and measuring the resulting depth distributions using SIMS. We obtained some undoped and some Al-doped ZnSe, and some undoped ZnTe from France. We included some of our own CdTe (obtained originally from SBRC). We performed mass surveys of these materials using SIMS to determine what impurities are present and in what approximate densities. Some of the ZnTe samples had been treated with a  $^2\text{H}$  plasma. We measured the resulting depth distributions of  $^2\text{H}$  using SIMS. We implanted the undoped and Al-doped samples of ZnSe with  $^2\text{H}$  and annealed pieces of these at temperatures of 300, 400, and 500°C, and measured the resulting depth profiles using SIMS.

More work for other annealing temperatures was prevented by the small amount of materials available. Some of these results are summarized below. For the undoped sample, at 400°C the  $^2\text{H}$  profile moved deeper into the ZnSe and did not disappear. For the 500°C anneal, the  $^2\text{H}$  density decreased some and the profile moved even deeper into the ZnSe material. Clearly, the behavior of H in ZnSe varies significantly depending on whether or not the material is doped (with Al in this case).

Before we received the ZnTe samples from France, they had been treated with a  $^2\text{H}$  plasma and had been profiled for  $^2\text{H}$ . We also profiled these samples for  $^2\text{H}$  and obtained agreement with the previous profiles, but obtained substantially greater detection sensitivity, which allowed us to measure better the details of the  $^2\text{H}$  at lower impurity densities, and with better depth resolution. Two or three growth interfaces could be measured in our analyses (two in one sample and three in the other sample).

## University Support

During the course of this contract work, we collaborated with research groups and university professors and their graduate students and post doctoral fellows. These collaborations are listed below. (9 universities and about 55 personnel)

Hampton University: Prof. U. Hoemmerich, J.T. Seo, M. Thaik, X. Wu, G. Ofori-Boadu, J. Prejean

Kansas State University: Prof. H. X. Jiang, R.A. Mair, C.J. Ellis, J.Y. Lin

Lehigh University: M.G. Weinstein, C.Y. Song, Prof. M. Stavola C.Y. Song

University of California at Santa Barbara: Prof. J. Speck and student Monica Hansen

University of Cincinnati: Prof. A. Steckl

University of Delaware: Prof. J. Kolodzey, Post-doc Cyril Guedj, M. Dashiell, G. Katulka C. Swann, M.W. Tsao, J. Rabolt

University of Texas at Austin: Prof. R. Dupuis, P.A. Grudowski

University Illinois: Prof. S. Bishop

University Florida: Professors S.J. Pearton, C. Abernathy, and F. Ren, and M. Overberg, X.A. Cao, R.K. Singh, M. Fu, J.A. Sekhar, J.D. MacKenzie, H.J. Guo, S.J. Pennycook, V. Scarvepalli, J.R. LaRoche, J.R. Lothian, J.W. Lee, D. Johnson, C.-M. Lee, C.-C. Chuo, G.-C. Chi, G.T. Dang, A.P. Zhang, S.N.G. Chu, L. Zhang, S.M. Donovan, H. Cho, K.B. Jung, R.F. Kopf, C.B. Vartuli, W.S. Hobson, M.M. Mshewa, J.-I. Chyi  
This has been a major collaboration - 31 persons and many papers.

## Meetings with ARO personnel

Because John Zavada was located in London during the time of this contract work, communication between Wilson and Zavada was carried out via email whenever appropriate (about once each month) and by fax or phone about once every two months. Zavada and Wilson did meet in Boston at the MRS meeting there during 2-4 Dec 1999. All aspects of the this contract (past, present, and future) were discussed. Several days of face-to-face meetings were held early in June, 2000, in Strasbourg France during the EMRS (European Materials Research Society) conference. Again, all aspects of the program were discussed - data, collaborations, papers, past work, and work to be done. Other collaborators were also present at the Strasbourg meeting and productive joint discussions of past and future work were held. Javada came to California to meet with Wilson once, but was called to Washington DC before this meeting could occur. There were many subsequent conversations via telephone and facsimile.

## Publications that resulted from this program

Many of the technical details of the work that was performed under this contract are described in the 50 references listed below, published during the years 1998, 1999, and 2000, the duration of this contract.

- S.J. Pearton, J.W. Lee, C.R. Abernathy, R.G. Wilson, J.M. Zavada, and J.C. Zolper, "Electrical and optical properties of C, O, and H in III-nitrides," *Electrochem. Soc. Proc.* **97-34**, 209 (1998)
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